Simple Fabrication of Metal-Based Piezoelectric MEMS by Direct Deposition of $Pb(Zr, Ti)O_3$ Thin Films on Titanium Substrates

Kensuke Kanda, Isaku Kanno, Hidetoshi Kotera, and Kiyotaka Wasa

Abstract*—Piezoelectric $Pb(Zr, Ti)O_3$ (PZT) thin films were directly deposited on cantilever-shaped titanium substrates and evaluated for their piezoelectric properties and actuator performance. Because of the small difference in the thermal expansion coefficient between the PZT and the substrate, and the mitigation of the residual stress, large piezoelectric properties could be obtained for PZT/Ti unimorph actuators. X-ray diffraction measurements clearly revealed that the PZT thin films have a polycrystalline perovskite structure with a random orientation. Observations using a scanning electron microscope (SEM) demonstrated that PZT films, which were 3.8 μm thick, were densely deposited on Pt-coated Ti substrate without pores or cracks. The polarization–electric field (P–E) hysteresis of the PZT film clearly indicates ferroelectricity. The piezoelectric properties of the PZT films were evaluated from the tip displacement of PZT/Ti unimorph cantilevers. Simplified transverse piezoelectric coefficients ($e^*_{31} = d_{31}/s^E_{11}$, where d_{31} and s^E_{11} are piezoelectric coefficient and elastic compliance, respectively) were measured, which ranged from -3.6 to 4.3 C/m^2—about three times larger than those of the PZT thin films deposited on stainless-steel substrates. Measurement of resonant frequencies of the cantilevers shows a clear dependence on the cantilever length, which obeys the theoretical equation. This indicates that these cantilevers can be reliably applied as sensors and actuators in a resonance mode. [2008-0100]

***Index Terms*—Microactuators, $Pb(Zr, Ti)O_3$ (PZT) ceramics, piezoelectricity, thin-film devices.**

I. Introduction

LEAD zirconate titanate [$Pb(Zr, Ti)O_3$: PZT] is widely used in various fields because of its excellent piezoelectric characteristics, which include high response rate, low energy consumption, and large displacement. PZT thin films, in particular, have been used in microelectromechanical systems (MEMS) for the integration of the piezoelectric function in actuators [1], [2], sensors [3], [4], and energy-harvesting devices [5], [6]. The deposition of PZT thin films is usually realized using sol–gel [7], [8], sputtering [9], and chemical vapor deposition [10]. Typical MEMS devices with a PZT thin film use a Si substrate, and the 3-D microstructure is realized by surface or bulk micromachining techniques in various geometries, such as membranes, bridges, and cantilevers. Si is the most common and useful substrate in MEMS because of its high elastic strength and the well-established Si microfabrication technologies. On the other hand, Si is brittle and exhibits no plastic deformation compared with metals [11]. Handling PZT thin films on metal substrates is much easier than on Si substrates. Furthermore, adhesion of PZT films in the field on Ti substrates is very strong, and peeling rarely occurs. Suzuki *et al.* [12] deposited PZT directly on cantilever-shaped stainless-steel substrates using radio frequency (RF) magnetron sputtering. This process realized simple fabrication of piezoelectric MEMS by using metal substrates free from the etching process of PZT, electrodes, and substrates obtained after PZT deposition. However, the transverse piezoelectric properties obtained were relatively low compared to those of the PZT films on other substrates, such as Si and MgO. This was probably due to the following: 1) polycrystalline perovskite structures with a preferential (1 0 1) orientation and 2) large compressive residual stress due to the mismatch of thermal expansion coefficients between PZT and stainless steel.

This paper addresses improvements in the piezoelectric properties of PZT thin films, which are directly deposited on the microfabricated substrate of metals. The deposition of the PZT films was conducted by RF magnetron sputtering because it would be very difficult to prepare PZT films with a uniform thickness by means of the sol–gel method on such microfabricated substrate. Furthermore, we substitute Ti substrates for stainless steel to improve the piezoelectric properties in this study. Ti has a thermal expansion coefficient close to that of PZT, which reduces the residual stress after PZT deposition. In this paper, we describe the piezoelectric properties of the PZT films on the Ti cantilever and their actuator performance.

II. Preparation

PZT thin films were directly deposited on 50-μm-thick Ti substrates, which had been microfabricated into the shape of a cantilever array by wet etching of a hydrofluoric acid solution [13]. Prior to the PZT deposition, Pt bottom electrodes (150 nm) and $(Pb, La)TiO_3$ (PLT) buffer layers (40 nm) were deposited on the Ti substrates by RF magnetron sputtering at a substrate temperature of about 600 °C. PLT has a perovskite structure with the lattice parameter $a = 0.391$ nm [14], which is compatible with the lattice parameter of PZT (i.e., $a = 0.402$ nm) [15], and it acts as a seed layer for the PZT growth [16]. PZT was subsequently deposited on PLT/Pt/Ti

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K. Kanda is with the Exploratory Research for Advanced Technology Project, Japan Science and Technology Agency (JST ERATO), University of Hyogo, Hyogo 671-2280, Japan (e-mail: kensuke.kanda@gmail.com).

I. Kanno, H. Kotera, and K. Wasa are with the Department of Micro Engineering, Kyoto University, Kyoto 606-8501, Japan.

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TABLE I
CONDITIONS FOR DEPOSITION OF PZT THIN FILMS

Parameter	Conditions
Substrate	PLT/Pt/Ti
Target	$[Pb(Zr_{0.53},Ti_{0.47})O_3]_{0.8}+[PbO]_{0.2}$
Substrate Temperature (°C)	600
Gas composition	Ar(19.5SCCM)+O_2(0.5SCCM)
Gas pressure (Pa)	0.33
RF power (W)	200
Anneal time (min)	60
Anneal temperature (°C)	650

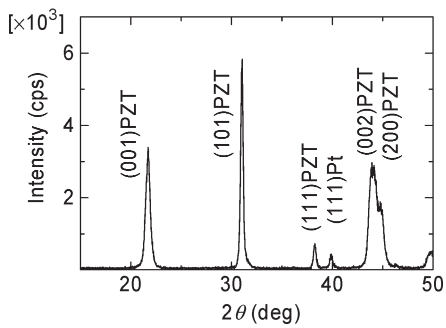

Fig. 1. XRD pattern of PZT thin-film deposited on a Pt-coated Ti cantilever array. A PLT seed layer is inserted between the interface of PZT and the Pt/Ti substrate.

by RF magnetron sputtering. For simplicity, in the following, PZT/PLT/Pt/Ti is referred to as PZT/Ti. The target PZT material for the sputtering was sintered ceramic with a composition of Zr/Ti $= 53/47$, which corresponds to the morphotropic phase boundary between the rhombohedral and tetragonal phases. The target included additional PbO to compensate for reevaporation of Pb from the growing film. The growth of the PZT thin film was carried out at a substrate temperature of about 600 °C in an Ar/O_2 mixed-gas atmosphere. The sputtering conditions are shown in Table I. For the actuator applications, we prepared relatively thick PZT films of 3.8 μm on the Ti substrates. After deposition, the samples were annealed at 650 °C for further crystallization of the PZT films.

III. MEASUREMENTS

A. Crystalline Structure

The crystalline structure of the PZT thin films was measured using X-ray diffraction (XRD, Rigaku Multi-flex). Fig. 1 shows the $2\theta/\theta$ XRD pattern of the PZT thin film deposited on a Ti substrate. There are several peaks for the perovskite phases of (0 0 1), (1 0 1), (1 1 1), and (0 0 2) PZT, and there is no heterogeneous phase, such as a pyrochlore structure. This indicates that the PZT films have a polycrystalline structure with random orientation. It should be noted that the PLT buffer layer was very effective to grow the pyrochlore-free PZT films on Ti substrates.

Fig. 2 shows scanning electron microscope (SEM) images of the obtained PZT surface and its cross section. From the surface SEM image, it is clear that the PZT consists of polycrystalline

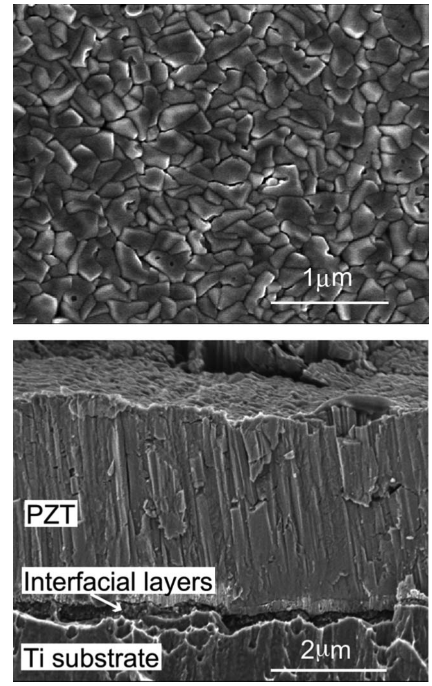

Fig. 2. SEM images of a PZT/Ti cantilever structure. (a) Surface. (b) Cross section. Dense and columnar grain structures are observed. The interfacial layer would contain Pt, PLT, and reaction layer.

grains of about 0.1–0.3 μm. Because Ti shows large plastic deformation, it was difficult to obtain a clear cleaved cross section of PZT/Ti. However, the cross-sectional SEM image indicates a dense columnar grain structure of the PZT thin film with a uniform thickness, and no pores or pin holes were observed in the films.

B. Electric Properties

The dielectric properties of the PZT thin film were measured using an inductance–capacitance–resistance meter. Au/Cr circular top electrodes having a diameter of 0.5 mm were deposited on the PZT thin films. The relative dielectric constant ε_r and the range of the dielectric loss $\tan\delta$ were determined to be about 506 and 0.01–0.02 at 1 kHz, respectively. Although relative dielectric constant ε_r of PZT/Ti is lower than the typical value of the PZT/Si, it is still higher than that of the PZT on stainless steel $(\varepsilon_r = 101)$ [12]. The P–E hysteresis loop, measured using the Sawyer–Tower circuit, is shown in Fig. 3. The deposited PZT thin films showed clear ferroelectricity with remanent polarization P_r of 20 $\mu C/cm^2$ and coercive electrical field E_c values of -50 and 70 kV/cm. These results show that PZT films on Ti substrates possess typical dielectric and ferroelectric properties, which are important characteristics for piezoelectric applications.

C. Piezoelectric Properties

To evaluate the piezoelectric properties, Ti sheets were fabricated into the shape of microcantilever arrays on which Pt, PLT, and PZT were deposited. The optical image of a piezoelectric cantilever array composed of PZT/Ti is shown in Fig. 4. The cantilevers ranged in length from 0.7 to 1.25 mm, with a width

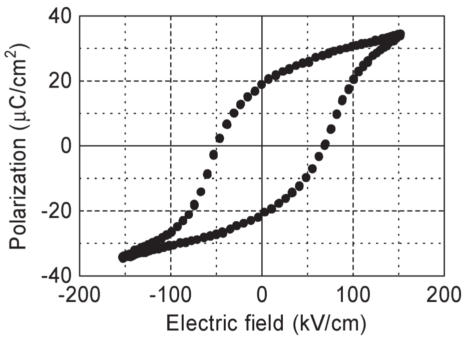

Fig. 3. P–E hysteresis loop of a PZT thin film measured at a frequency of 1 kHz. Clear ferroelectric behavior is confirmed.

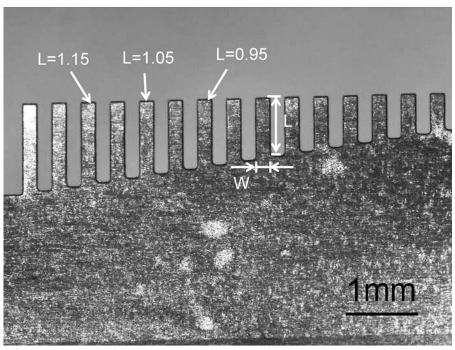

Fig. 4. Optical image of a PZT/Ti cantilever array.

TABLE II
DIMENSIONS OF CANTILEVERS

Thickness (μm)	
Ti substrate	50
Pt electrode	0.15
PLT buffer layer	0.04
PZT layer	3.8
Length (mm)	0.7-1.25
Width (mm)	0.2

of 0.2 mm. The dimensions of the cantilevers are given in Table II. The gold on chromium (Au/Cr) top electrode was deposited on the PZT films, and the actuator characteristics were evaluated from the tip displacement of the cantilever, measured using a laser Doppler vibrometer (Graphtec AT-3700) and a laser interferometer (Graphtec AT-1200). The top Au/Cr electrode covers the whole top surface of PZT and the lower electrode consists of Ti substrates, and the electrodes were wired at a fixed position in the substrate. Prior to the measurements, we applied a voltage of -30 V on the top electrode whose electric field is higher than the coercive electric field observed in Fig. 3. It makes the polar direction align as the surface of the PZT is positive. The PZT/Ti did not need intensive poling treatment like bulk PZT because it had very stable polarization, and the application of the electric field just higher than E_c was enough to obtain the stable piezoelectric property. The stable polarization and piezoelectricity for the PZT films prepared by RF sputtering were reported in previous studies [17], [18].

Fig. 5 shows a time-series of the cantilever deflection when applying a negative unipolar sine-wave voltage at a frequency

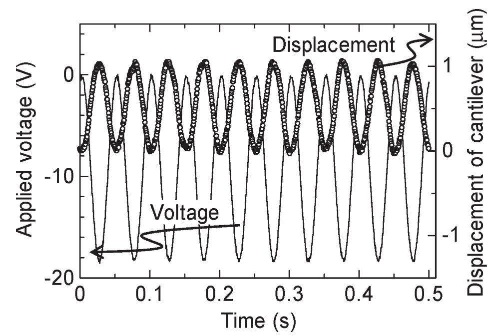

Fig. 5. Time-series variation of the input signal and tip deflection of a cantilever when the cantilevers vibrate at a frequency of 20 Hz. Clear piezoelectric vibrations could be obtained.

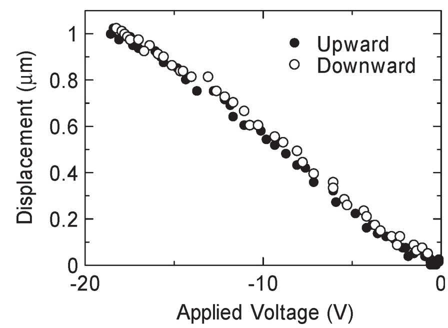

Fig. 6. Tip displacement as a function of applied voltage. There is a small amount of hysteresis.

of 20 Hz, measured with a laser interferometer. The displacement phase was reversed from the input voltage. The application of negative voltage in the polar direction of the PZT causes shrinkage of the PZT thin films along the length due to the transverse piezoelectric effect, resulting in an upward displacement. This means that the cantilever vibration was induced by a piezoelectric effect, and not by a thermal effect, whose displacement should have the same phase as the input signal.

Hysteresis of the tip displacement was observed using the laser interferometer while applying one cycle of the negative sine-wave voltage at a frequency of 20 Hz. The result is shown in Fig. 6. The displacement hysteresis was very small, indicating that the linear control of the cantilever deformation would be possible. The relationship between tip displacement of cantilevers having lengths of 0.95, 1.05, and 1.15 mm and the applied electric fields was evaluated by applying a unipolar sine-wave signal at a frequency of 1 kHz, as shown in Fig. 7. Tip displacement was proportional to the applied voltage.

We evaluated the transverse piezoelectric properties by measuring the cantilever displacement. The piezoelectric properties of d- and e-forms are expressed by the following equations:

$$T_i = c_{ij}^E S_j - e_{ki} E_k \tag{1}$$

$$S_i = s_{ij}^E T_j - d_{ki} E_k \tag{2}$$

where $k = 1, 2, 3$ and $i, j = 1, 2, 3, 4, 5, 6$. T_i, S_j, and E_k are the stress, strain, and electric field components, c_{ij}^E and s_{ij}^E are constant-electric-field elastic stiffness and elastic compliance components, and e_{ki} and d_{ki} are piezoelectric coefficients [2]. For a piezoelectric thin-film form deposited on the substrate,

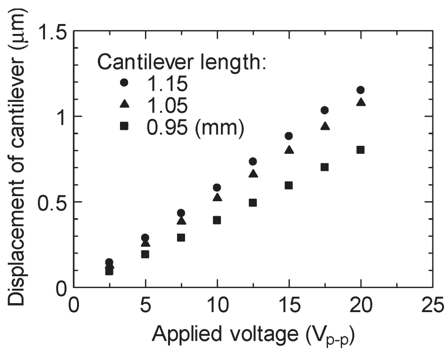

Fig. 7. Relationship between the applied voltage and tip displacement for cantilevers that are 0.95, 1.05, and 1.15 mm long.

we can assume in-plane and shear strains to be zero. If we apply an external electric field along the thickness, the transverse piezoelectric coefficient e_{31} is written as

$$e_{31} = \frac{c_{13}^E S_3 - T_1}{E_3} = \frac{c_{13}^E S_3}{E_3} + \frac{d_{31}}{s_{11}^E + s_{12}^E}. \tag{3}$$

Since the effective transverse piezoelectric coefficient $e_{31,f}$ is defined as $e_{31,f} = d_{31}/(s_{11}^E + s_{12}^E)$ [19], (3) can be expressed as

$$e_{31} = e_{31,f} + \frac{c_{13}^E}{c_{33}^E} e_{33}. \tag{4}$$

On the other hand, the tip displacement of the unimorph cantilever is expressed as

$$\begin{aligned} d_{31} &= -\frac{\delta K}{3 s_{11,p}^E s_{11,s} h_s (h_s + h_p) V L^2} \\ K &= 4 s_{11,p}^E s_{11,s} h_s (h_p)^3 + 4 s_{11,p}^E s_{11,s} (h_s)^3 h_p + \left(s_{11,p}^E\right)^2 (h_s)^4 \\ &\quad + \left(s_{11,s}^E\right)^2 (h_p)^4 + 6 s_{11,p}^E s_{11,s} (h_s)^2 (h_p)^2 \end{aligned} \tag{5}$$

where h, s_{11}, L, V, and δ are the thickness, elastic compliance, cantilever length, applied voltage, and tip displacement, respectively [20]. The subscripts "s" and "p" denote the substrate and PZT thin film, respectively. When the thickness of the PZT thin film is negligible compared to that of the substrate, the transverse piezoelectric coefficient d_{31} can be estimated as

$$d_{31} \cong -\frac{h_s^2}{3L^2} \frac{s_{11,p}^E}{s_{11,s}} \frac{\delta}{V}. \tag{6}$$

The mechanical properties of the thin films influence the deposition process and the substrates; therefore, it is critical to measure the elastic properties of the PZT thin films precisely. However, they are difficult to be identified. In this paper, we evaluated the piezoelectric properties of the PZT thin films on Ti substrates using a simplified transverse piezoelectric coefficient to eliminate the ambiguous material properties of the thin films. It is given as

$$e_{31}^* = \frac{d_{31}}{s_{11,p}^E} \cong -\frac{h_s^2}{3 s_{11,s} L^2 V} \delta. \tag{7}$$

The simplified transverse piezoelectric coefficient e_{31}^* is shown in Fig. 8. The e_{31}^* of the PZT films on Ti substrates ranged from -3.6 to -4.3 C/m^2 and exhibited an almost constant value of up to 53 kV/cm over the external electric fields. This was due to the clear linearity between the applied voltage and displacement. Assuming that the s_{11} of the PZT film is the same as that of PZT-8 ($s_{11} = 11.1 \times 10^{-12}$ m^2/N) [21], d_{31} of PZT/Ti is estimated as -40.0 to -47.7 pm/V. These results indicate that the e_{31}^* of the PZT films on Ti substrates was up to three times that of stainless-steel substrates [12] and was almost comparable to the PZT films on Si or MgO [17]. This implies that a great improvement in the piezoelectric characteristics was realized by replacing stainless-steel substrates with Ti substrates. The improvement of the piezoelectricity is attributed to the pyrochlore-free PZT films on Ti substrates. The Ti substrates may also be advantageous to enhance the piezoelectricity due to the small internal stress induced by the difference of the thermal expansion coefficients of PZT and Ti, e.g., 8.0×10^{-6} °C^{-1} for PZT, 8.6×10^{-6} °C^{-1} for Ti, and 17.2×10^{-6} °C^{-1} for stainless steel [22]. Note that Pt and PLT buffer layers have been used in stainless-steel substrates in [12].

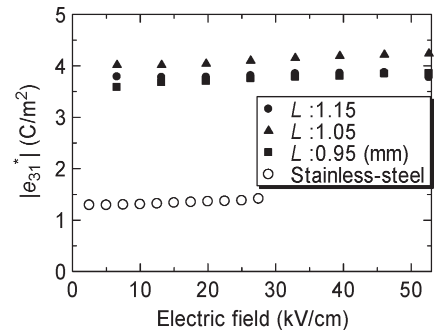

Fig. 8. Simplified transverse piezoelectric coefficient measured in this study. The dots for stainless steel represent conventional values [12].

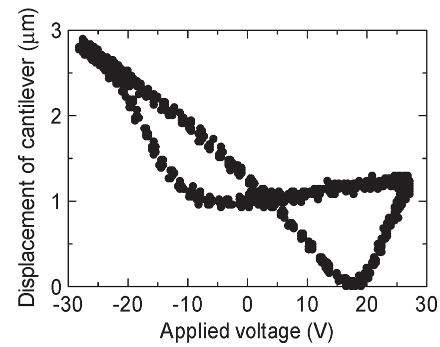

Fig. 9. Tip deflection of the cantilevers when applying a bipolar sine wave voltage at a frequency of 20 Hz. A typical butterfly shape is observed.

To investigate the domain motion of the PZT thin films on the Ti cantilevers, tip displacement was measured using a laser interferometer, applying bipolar sine-wave voltages at 20 Hz. Fig. 9 shows the tip deflection of the unimorph cantilever. A clear butterfly-shaped curve was observed, with polarization reversals at electric fields of $+20$ and -10 kV/cm.

D. Oscillation Characteristics

To evaluate the performance of cantilever arrays as actuators, oscillation characteristics were measured as a function of frequency. The frequency response of cantilevers having lengths

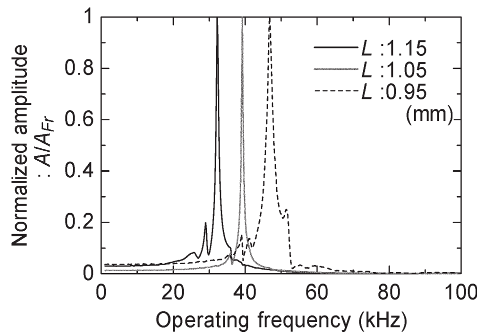

Fig. 10. Frequency response of PZT/Ti cantilevers having different lengths (i.e., 1.15, 1.05, 0.95 mm). An independent resonant frequency is clearly observed for each cantilever. The vertical axis indicates normalized amplitude, where A_{Fr} and A are amplitude at resonant frequency and amplitude, respectively.

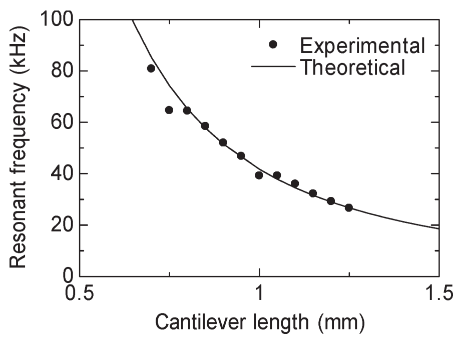

Fig. 11. Relationship between cantilever length and resonant frequency. The resonant frequency for each cantilever obeys the theoretical equation.

0.95, 1.05, and 1.15 mm were measured using a laser Doppler vibrometer. Fig. 10 shows the normalized amplitude of the vibration for each cantilever as a function of frequency. The small satellite peaks were observed adjacent to each main peak due to the imperfection of fixed edge of the cantilever. However, the resonant frequency of each cantilever obeyed the following theoretical equation:

$$f_r = \frac{1}{2\pi}\left(\frac{1.875}{L}\right)^2 \sqrt{\frac{EI}{\rho A}} \tag{8}$$

where E, I, ρ, and A are the elastic constant, second moment of area, density, and cross-sectional area, respectively. The first resonant frequencies for the cantilevers with lengths ranging from 0.7 to 1.25 mm are plotted in Fig. 11. By using Ti properties of Young's modulus $E = 104$ GPa and density $\rho = 4510\ \mathrm{kg/m^3}$ [22], the experimental results closely correspond with the calculated values, implying that all cantilevers in the array can be independently operated without interaction. In addition, it assures the effectiveness of applying the cantilever array to sensors and actuators using resonant oscillation.

IV. Conclusion

To provide both high piezoelectric performance and simple fabrication of piezoelectric MEMS, PZT thin films were directly deposited on a titanium cantilever array, and the crystalline, ferroelectric, piezoelectric, and oscillation characteristics were evaluated. The PZT thin films were deposited using RF sputtering; the resulting films exhibited polycrystalline perovskite structures with random orientation. The P–E hysteresis loops indicated typical ferroelectric behavior. The piezoelectric properties of the PZT films were evaluated from the tip displacement of PZT/Ti unimorph cantilevers, and the simplified transverse piezoelectric coefficient e_{31}^* (d_{31}/s_{11}^E) was calculated, which ranged from -3.6 to $-4.3\ \mathrm{C/m^2}$. The enhancement of piezoelectric properties compared to PZT films on other metal substrates, such as stainless steel, is attributed to the fact that the thermal expansion coefficients of PZT and Ti are compatible. Although we have not confirmed the evidence of the interdiffusion between PZT and substrate materials, Ti is a component of PZT and compositional affinity might be effective to obtain higher piezoelectric properties than that of PZT/stainless steel. Measurements of the resonant characteristic of the PZT/Ti cantilevers showed a clear dependence on the cantilever length, which obeys the theoretical equation. This assures that the cantilevers can be reliably applied as sensors and actuators in MEMS.

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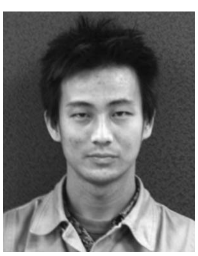
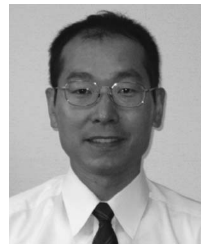


Kensuke Kanda received the B.E., M.E., and Dr.Eng. degrees in mechanical engineering from Tokyo Metropolitan University, Tokyo, Japan, in 2000, 2003, and 2006, respectively.

From 2006 to 2008, he was a Postdoctoral Researcher with Advanced Software Technology and Mechatronics Research, Institute of Kyoto, Kyoto, Japan. In 2008, he joined the Exploratory Research for Advanced Technology Project, Japan Science and Technology Agency (JST ERATO), University of Hyogo, Hyogo, Japan. His research interests include integration of sensors and actuators, near-surface fluid dynamics, and development of microfluidic devices, such as micropumps, microvalves, and micromixers.



Isaku Kanno was born in Fukuoka, Japan, in 1966. He received the B.S., M.S., and Ph.D. degrees from Osaka University, Osaka, Japan, in 1989, 1991, and 1999, respectively.

From 1991 to 2002, he was with the Research and Development Center, Panasonic Inc., Osaka. From 2002 to 2005, he was an Associate Professor with the Department of Mechanical Engineering, Kyoto University, Kyoto, Japan, where he has been an Associate Professor with the Department of Micro Engineering since 2002. His current research interests include growth of ferroelectric thin films, characterization of the relationship between crystalline structure and piezoelectric properties of films, and application of piezoelectric films to MEMS devices.

Hidetoshi Kotera was born in Osaka, Japan, in 1957. He received the B.E., M.E., and Dr.Eng. degrees in mechanical engineering from Kyoto University, Kyoto, Japan, in 1980, 1982, and 1992, respectively.

From 1982 to 1993, he was a Research Manager with Matsushita Electric Industrial Co. Ltd., Osaka, Japan. At the central research laboratory, he engaged in research involving numerical analysis of electromagnetics, fluid dynamics, structural analysis, and molecular dynamics. He was with the Department of Mechanical Engineering, Kyoto University, from 1993 to 2001 as an Associate Professor and from 2001 to 2005 as a Professor. Since 2005, he has been with the Department of Micro Engineering, Kyoto University, as a Professor. He is currently involved in MEMS, micro-TAS, and computational science research at Kyoto University.

Kiyotaka Wasa received the B.S. degree and the Ph.D. degree in plasma physics from Osaka University, Osaka, Japan in 1960 and 1968, respectively.

In 1960, he joined Panasonic and studied plasma in a magnetron discharge until 1990. From 1990 to 1998, He was Deputy Director of the Research Institute of Innovative Technology for the Earth (RITE) of Japan. From 1988 to 2003, he was with Yokohama City University, Yokohama, Japan, as a Professor. He is currently with the Department of Micro Engineering, Kyoto University, Kyoto, Japan, as a Japan Science and Technology Agency Researcher and with the University of Electronic Science and Technology, Chengdu, China, and Nanjing University, Nanjing, China, as an Honorary Professor. He has performed seminal work on magnetron sputtering and has developed numerous thin-film materials and electronic devices, including ZnO, diamonds, and high-T_c superconductors.